JOAP-TSC-TR-06-04

OPTIMIZATION OF A STABINGER VISCOMETRIC METHOD TO MAXIMIZE SAMPLE THROUGHPUT

Balancing the Competing Interests of Speed, Cost, and Data Quality

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14. ABSTRACT

The U.S. Army Oil Analysis Program has sought to purchase automated viscometers (with autosamplers) for its centralized laboratories. The Army OAP has a sample throughput requirement of at least 40 samples per hour. In principle, this is within reach of the Anton Paar SVM3000 Stabinger viscometer. In addition, anecdotal evidence in our laboratory had led to disagreement regarding flush volume and measurement time requirements for optimal and sufficient performance of the SVM3000. Therefore, an investigation of the Stabinger viscometer was carried out to provide a scientific basis for parameter optimization while simultaneously balancing the competing requirements for speed, cost, and data quality. As a result of this investigation, it has been determined that the rigors of ASTM standard D 7042-04 exceed the programmatic requirements of the Army OAP. The ASTM standard biases the golden triangle (good, fast, cheap) in favor of higher data quality and sacrifices the competing interests of cost and speed. It has been determined that measurement duration (stabilization time) can be set to 60 seconds while meeting Army OAP programmatic needs for data quality, but the extant firmware does not permit this. In addition, this study showed that flush volumes of 20.0 mL satisfactorily purge and recondition the densimeter and viscometer measuring chamber without external washes between the analyses. This practice yields data of sufficient quality for Army OAP decision-making. This flush volume requires the 24-position carousel for the autosampler, which uses 50 mL sample vials. A manually operated SVM series Stabinger viscometer can achieve or exceed the required Army OAP sample throughput when injecting with a 10 mL syringe, but an autosampler-equipped SVM series Stabinger viscometer is unable to do so with the current firmware configuration. Nevertheless, a revised configuration that is currently under development is anticipated to provide flexibility sufficient for the recommendations made in this report to be adopted. It is also recommended that dynamic viscosity replace kinematic viscosity as the main indicator of oil quality and that a separate oil density limit be instituted either implicitly via the co-adoption of the kinematic viscosity or explicitly as a permissible or acceptable density range.

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/s/

Daniel A. Jensen
Major, United States Army
Director
Joint Oil Analysis Program
Technical Support Center

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OPTIMIZATION OF A STABINGER VISCOMETRIC METHOD TO MAXIMIZE SAMPLE THROUGHPUT

Balancing the Competing Interests of Speed, Cost, and Data Quality

Executive Summary

The U.S. Army Oil Analysis Program has sought to purchase automated viscometers (with autosamplers) for its centralized laboratories. The Army OAP has a sample throughput requirement of at least 40 samples per hour. In principle, this is within reach of the Anton Paar SVM3000 Stabinger viscometer. In addition, anecdotal evidence in our laboratory had led to disagreement regarding flush volume and measurement time requirements for optimal and sufficient performance of the SVM3000. Therefore, an investigation of the Stabinger viscometer was carried out to provide a scientific basis for parameter optimization while simultaneously balancing the competing requirements for speed, cost, and data quality. As a result of this investigation, it has been determined that the rigors of ASTM standard D 7042-04 exceed the programmatic requirements of the Army OAP. The ASTM standard biases the golden triangle (good, fast, cheap) in favor of higher data quality and sacrifices the competing interests of cost and speed. It has been determined that measurement duration (stabilization time) can be set to 60 seconds while meeting Army OAP programmatic needs for data quality, but the extant firmware does not permit this. In addition, this study showed that flush volumes of 20.0 mL satisfactorily purge and recondition the densimeter and viscometer measuring chamber without external washes between the analyses. This practice yields data of sufficient quality for Army OAP decision-making. This flush volume requires the 24-position carousel for the autosampler, which uses 50 mL sample vials. A manually operated SVM series Stabinger viscometer can achieve or exceed the required Army OAP sample throughput when injecting with a 10 mL syringe, but an autosampler-equipped SVM series Stabinger viscometer is unable to do so with the current firmware configuration. Nevertheless, a revised configuration that is currently under development is anticipated to provide flexibility sufficient for the recommendations made in this report to be adopted. It is also recommended that dynamic viscosity replace kinematic viscosity as the main indicator of oil quality and that a separate oil density limit be instituted either implicitly via the co-adoption of the kinematic viscosity or explicitly as a permissible or acceptable density range.

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1. Introduction

1.1. Background

At the request of the U.S. Navy Oil Analysis Program (OAP), the JOAP TSC previously undertook an investigation of commercial viscometers (*1*). That investigation concluded that the Anton Paar Stabinger viscometer provided superior dynamic viscosity data, closely followed by the Cambridge viscometer. The Stabinger viscometer's operating principle is that the viscosity of a fluid can be determined by the drag the fluid exerts on a free-spinning rotor. The outer tube containing the fluid spins and the viscous forces transfer the rotational motion to the inner rotor, which contains a magnet. The magnet permits the measurement of the rotational velocity via the Hall effect. This has been described in detail elsewhere (*2*). Based on the manufacturer's guidance, the JOAP TSC used a streamlined method for the measurement of viscosity on the Stabinger viscometer. Due to the convenience, ease of use, accuracy, and precision associated with the Anton Paar viscometer, the JOAP TSC purchased an SVM3000 for the laboratory and has used it throughout the past two years without any major problems.

Subsequent to the conclusion of our investigation, Anton Paar sought to have a written method for the Stabinger viscometer approved by the American Society for Testing and Materials. In fact, the JOAP TSC participated in a multiple laboratory validation in support of the ASTM petition. ASTM D 7042 was eventually approved by Subcommittee D02.07 and the American National Standards Institute (*3*). ASTM D 7042 requires an extensive washing and drying cycle between samples. This cycle leads to relatively long analysis times pre sample.

Despite the existence of the ASTM standard, the JOAP TSC has continued to use the procedure established during its first investigation of the Anton Paar SVM3000. This procedure permits the analysis of an individual sample in approximately two minutes, with an average sample throughput rate of about 30 per hour. The rate may be higher, but this varies with the nature of the sample. During this two-year period, JOAP TSC staff have continually made several observations regarding performance of the SVM3000. Anecdotally, it has been reported by JOAP TSC staff that two 10-mL injections of sample tended to give better results than a single 10-mL injection. In addition, it has been noted that the viscosity displayed by the instrument tends to remain reasonably fixed (2-3 significant digits) after a relatively short period of time, sometimes as little as 30 seconds.

Recently, the U.S. Army Oil Analysis Program has sought to purchase automated viscometers (with autosamplers). The Army OAP has a sample throughput requirement of at least 40 samples per hour (*4*). The Army OAP has determined that this throughput ensures cost-effectiveness of the test and result. During a recent demonstration by Anton Paar sales staff at Redstone Arsenal, this requirement was not met using a set of samples provided to the Anton Paar techincian by the Army OAP run per ASTM D 7042-04.

1.2. Aim of Investigation

Given the Army requirement and the anecdotal evidence of our own laboratory, it was deemed prudent to further investigate the Stabinger viscometer and provide a scientific basis for measurement parameters. In addition, we recognize that ASTM D 7042-04 is intended to provide high quality data, perhaps a higher quality than required for the task at hand. Thus, it seems we once again find ourselves face-to-face with a premier mantra of analytical chemistry: *Good, fast cheap–pick two.*

Accordingly, we set out to accomplish two objectives: to determine the flush volume and measurement duration for optimal results and to determine the impact of using suboptimal flush volume and measurement duration on overall data quality. Strict adherence to ASTM D 7042-04 is not required for our purposes, but it is necessary to ensure that the data are of sufficient precision and accuracy to meet Army OAP needs.

2. Experimental section

2.1. Instrumentation and Reagents

An Anton Paar SVM 3000 Stabinger Viscometer was used throughout the experiment. The functional parts of the viscometer in contact with the oil were cleaned in kerosene and standards were run to confirm the accuracy of the viscometer. The lubricants are listed in Table 1. The manufacturers' seals were broken, and aliquots of approximately 500 mL were placed into polypropylene bottles for convenience and refilled as necessary.

Table 1. Lubricants used in this study

Code[a]	Manufacturer, item, lot/batch no.	DOD/MIL specification	Description	National stock number
RTO	Royco[b] Turbine Oil 555, lot no. L6010	DOD-L-85734	helicopter transmission fluid	9150-01-209-2684
PP30	Pitt Penn[c] 30 (HDO-30), batch no. 4L0598	MIL-PRF-2104G	engine lubricating oil	9150-01-178-4726
CGO	CSD[d] Gear Oil 75 (75W) (O-186), lot no. UC4L22W092	MIL-PRF-2105E	gear lubricating oil	9150-01-035-5390

Notes: (a) Code used to refer to the oil in this report. (b) Royal Lubricant Co. (c) Pitt Penn Oil Co., Creighton, PA 15030. (d) CSD, Inc., Tucker, GA.

2.2. Effect of Measurement Duration

A 10-mL aliquot of oil was introduced into the sample port on the viscometer with a disposable polypropylene syringe; this incorporates a flush volume and a sample volume. After a delay of ~ 5 s, the rotor was started; a timer was started immediately thereafter. Data were recorded at 30, 60, and 90 s and at completion. The final value (infinite time) is based on stability criteria within the firmware of the instrument and is usually close to 2 minutes, but can occasionally be longer than 3 minutes. These data permit the assessment of stability as a function of time. The viscometer reports four values: dynamic viscosity (η, expressed in mPa s), kinematic viscosity (μ, expressed in mm^2 s^{-1}), density (ρ, expressed in g cm^{-3}), and temperature (T, expressed in ˚C). The kinematic viscosity is calculated by the instrument directly and equals the ratio of dynamic viscosity to density, i.e., $\mu = \eta/\rho$.

2.3. Effect of Flush Volume

The three different oils were run in triplicate in a sequence described below to ascertain the effect of the sequence. Each injection incorporates a flush of the measurement chamber, using approximately 7 mL of the injected volume for flushing the chamber. Consequently, every subsequent replicate has the effect of an additional (~ 10 mL) flush volume; the third replicate will have had a flush of nearly 30 mL. The sequence was as follows (MIL/DOD specification follows the slash): RTO/85734 ➙ PP30/2104 ➙ CGO/2105 ➙ RTO/85734 ➙ CGO/2105 ➙ PP30/2104 ➙ RTO/85734. This provides all possible permutations: A/B, A/C, B/A, B/C, C/A, C/B.

3. Analysis, results, and discussion

3.1. General Observations

The raw and treated data provided by the viscometer are listed in Appendix 1. Summary statistics based on Appendix 1 data are given in Table 2. The viscometer uses fixed criteria for stability and these criteria yield the values reported as final or represented as occuring at infinite (∞) time in Table 2. The arithmetic mean, standard error (estimated standard deviation of the mean), are reported for four experimental values: dynamic viscosity (η, expressed in mPa s), kinematic viscosity (μ, expressed in mm^2 s^{-1}), density (ρ, expressed in g cm^{-3}), and temperature (T, ˚C). This permits the assessment of stability as a function of time.

For the individual data sets (triplicate measures), the geometric mean was always within 0.04% of the arithmetic mean, making it unnecessary to distinguish between these two measures of central tendency. The term *tightness* is used herein to refer to the ratio of the range to the arithmetic mean. When dealing with small sets of data (triplicate measures here), it is often most helpful to look at the difference between the maximum and the minimum with respect to the arithmetic mean. The utility of the estimated standard deviation increases as the size of the data set grows. The raw data are tight. Most of the data sets have a tightness less than 0.04 with all less than 0.07. A single outlier was

discarded of the 30-weight oil, presumably due to the presence of an air bubble in the densimeter that led to an aberrantly low density and subsequently a correspondingly low kinematic viscosity.

Table 2. Summary statistics for data on the SVM3000 at various measurement durations

		30 s		60 s		90 s		final (∞)	
Oil[a]	Statistic	η, mPa s	μ, mm^2 s^{-1}	η, mPa s	μ, mm^2 s^{-1}	η, mPa s	μ, mm^2 s^{-1}	η, mPa s	μ, mm^2 s^{-1}
RTO	mean[b]	28.03	28.42	27.06	27.68	26.92	27.61	26.86	27.59
	std error[c]	0.18	0.23	0.23	0.27	0.24	0.27	0.23	0.26
	range	1.464	1.671	1.664	1.854	1.701	1.854	1.603	1.813
	tightness[e]	0.05	0.06	0.06	0.07	0.06	0.07	0.06	0.07
PP30	mean	87.72	99.934	84.099	96.776	83.666	96.476	83.339	96.217
	std error	0.40	0.46	0.34	0.38	0.38	0.44	0.42	0.49
	range	2.480	2.763	2.280	2.621	2.519	2.859	2.741	3.168
	tightness	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03
CGO	mean	106.97	121.17	103.48	118.09	103.05	117.83	102.59	117.02
	std error	0.24	0.22	0.38	0.49	0.44	0.53	0.48	0.70
	range	1.25	1.40	2.50	3.20	2.82	3.38	3.09	3.61
	tightness	0.01	0.01	0.02	0.03	0.03	0.03	0.03	0.03

		30 s		60 s		90 s		final (∞)	
Oil	Statistic	ρ, g cm^{-3}	T, °C	ρ, g cm^{-3}	T, °C	ρ, g cm^{-3}	T, °C	ρ, g cm^{-3}	T, °C
RTO	mean	0.979	40.16	0.976	40.02	0.975	39.97	0.974	40.01
	std error	0.001	0.004	0.001	0.005	0.001	<0.001	0.001	<0.001
	range	0.008	0.030	0.008	0.045	0.008	0.007	0.007	0.003
	tightness	0.008	0.001	0.008	0.001	0.008	<0.001	0.007	<0.001
PP30	mean	0.87	40.17	0.87	39.99	0.867	39.97	0.866	40.02
	std error	<0.001	0.012	<0.001	0.005	<0.001	0.002	<0.001	0.002
	range	0.002	0.081	0.001	0.033	0.001	0.013	0.001	0.012
	tightness	0.002	0.002	0.001	0.001	0.001	<0.001	0.001	<0.001
CGO	mean	0.877	40.17	0.875	39.98	0.874	39.97	0.873	40.00
	std error	<0.001	0.008	<0.001	0.005	<0.001	0.002	<0.001	<0.001
	range	0.003	0.059	0.003	0.027	0.003	0.003	0.003	0.002
	tightness	0.004	0.001	0.003	0.001	0.003	0.005	0.003	<0.001

Notes: (a) RTO = Royco Turbine Oil 555 (DOD-L-85734), PP30 = Pitt Penn 30 (MIL-PRF-2104G), CGO = CSD gear oil 75 (MIL-PRF-2105E). (b) Mean = arithmetic mean of 9 measurements for RTO or 6 measurements for PP30 and CGO. (c) Standard error is a measure of error in the mean and is given by $s/\sqrt{n}$ where s is the estimated standard deviation and n is the number of replicates. (e) Tightness is the ratio of range to mean, a useful measure of spread for small sets of data.

3.2. Effect of Measurement Duration

In general, most mineral oils reasonably approximate Newtonian fluid behavior, which means that their viscosities are invariant to both the time a shearing force is applied and the rate of the shear. Instead, the viscosity of a Newtonian fluid depends only on temperature. Newtonian behavior is important for engine oils because the thickness of the lubricant film—and therefore the viscosity—must remain constant regardless of the applied shear stress or changes to it. During normal engine operation, shear rate undergoes rapid changes and shear stress is applied for long periods of time, but lubricant film thickness is required to be constant. An oil that could not maintain its viscosity would be unable to maintain lubricity as a result of changes in the film thickness. In addition, an oil whose viscosity changed with shear rate would adversely affect power output and would tax the engine under those conditions where viscosity was increased. Although an assumption of Newtonian behavior simplifies our task, strict Newtonian behavior is not required. So long as the fluids inside

the measuring chamber are miscible and reasonably free of time-dependent effects (rheopexy and thixotropy), the viscous properties will eventually stabilize if the measurement is carried on long enough. Shear-rate-dependent viscoelastic effects will eventually stabilize so that pseudoplasticity and dilatancy are not problematic even if they occur.

Essentially, what we are interested in here is not the viscosity itself, but the speed with which the viscometer response stabilizes. Accordingly, the nature of the individual samples is not so important, provided that any mixtures of fluids are entirely miscible, which we know to be the case. Moreover, it is sufficient to treat all the data at once since all the injections represent real samples. Aggregate statistics are shown in Table 3 for the relative difference between the values of η, μ, ρ, and T at intermediate measurement duration (i.e., 30, 60, or 90 s) and final time as determined by the fixed criteria of the viscometer's internal firmware.

Table 3. Fractional difference in dynamic viscosity, kinematic viscosity, density, and temperature relative to final value at various measurement durations on the SVM 3000

Time[a], s	Statistic[b]	$\Delta\eta/\eta_\infty$, %	$\Delta\mu/\mu_\infty$, %	$\Delta\rho/\rho_\infty$, %	$\Delta T/T_\infty$, %
30	mean	4.60	3.42	0.51	0.40
	extremum	6.66	5.76	0.59	0.51
60	mean	0.83	0.57	0.25	–0.02
	extremum	1.48	3.00	0.30	0.08
90	mean	0.34	0.31	0.11	–0.08
	extremum	0.67	2.85	0.14	–0.11

Notes: (a) Time refers to the measurement duration (time elapsed/allowed for stabilization). (b) The mean is the arithmetic mean of 18 replicate measurements under repeatability conditions. The extremum refers to the largest deviation (maximum of absolute values) for ΔP for any single injection. (c) For each property P, $\Delta P = P_t - P_\infty$; properties: η (dynamic viscosity in mPa s), μ (kinematic viscosity in $mm^2\ s^{-1}$), ρ (density in $g\ cm^{-3}$), and T (temperature in ˚C).

The viscometer begins the measurement with the fluid and rotor at rest, so that the viscosity appears to be infinite at the start of the measurement process. As the spinning rotor reaches its maximum angular velocity, the viscometer signal decreases, eventually remaining virtually fixed. In practice, we find that the dynamic viscosity progress from η_0 to η_∞ is entirely asymptotic. Density also seems to follow this pattern, but the difference is quite small between ρ_0 and ρ_∞. Accordingly, the kinematic viscosity must behave similarly. Temperature, on the other hand, can fluctuate somewhat around its final value, probably due to the interplay among the heater, the equilibrating (warming) fluid, and thermostat. Temperature stabilizes quickly, differing from its final value by no more than 0.5% after 30 s and no more than 0.1% at 60 s. Density stabilizes quickly as well, differing from its final value by no more than 0.6% after 30 s and no more than 0.3% after 60 s. Dynamic viscosity comes to within 7% of its final value after 30 s, but stabilizes to within 1.5% of its final value within 60 s. By 90 s, dynamic viscosity is less than 0.7% from its final value. The stabilization times vary to as long as 2-3 minutes owing to the precisional requirements of the firmware; nevertheless, it is possible to obtain satisfactory data by truncating the measurement at 90 s or even 60 s. Given the viscosity

ranges permissible for most military lubricants and keeping in mind that the viscosity measurement is always biased high when taken early, it would be possible to account for the difference in the limits. In other words, we can confidently say that there is no net benefit to be realized by the Army OAP in allowing longer than 60 s for stabilization to occur. It is concluded that strict adherence to ASTM D 7042-04 would not be beneficial, since it would sacrifice *fast* and *cheap* in favor of *good*. The improvement in data quality is not justifiable programmatically.

3.3. Effect of Flush Volume

Figure 1. Anton Paar SVM3000 viscometer is shown with Xsample 360 autoinjector

Most autosampler/autoinjector devices used in gas chromatography (GC) use a syringe that requires a wash cycle, often using different wash solutions for pre-injection and post-injection cleaning. On account of the highly volatile nature of the solvent in contrast with the sometimes rather involatile nature of the analytes or other solutes, this type of wash process is necessary to clean residual sample from the syringe and eliminate carryover to the next injection. Unlike GC autoinjectors, the injector port in the viscometer and the measurement chambers (densimeter and rotor cell) cannot be cleaned by heating. Most autosampler/autoinjector devices used in liquid chromatography (LC) use a sample loop rather than a syringe to measure out the sample. The sample loop is cleaned with either a wash solution, multiple volumes of the next sample, or a combination. Normally, most or all of the sample is consumed in this process. The Anton Paar autosampler-viscometer system is most similar to an LC autoinjector in that it requires that the next sample be used to wash out both the injection port and the measurement chamber. Unlike an LC system, it does not have columns or mobile phase, so the measurement chamber is thematically undifferentiated from the injector port. Accordingly, it is necessary that the process draw from the principles used for both of those autosampler/autoinjector types. Anton Paar has imposed wash and dry cycles in order to conserve sample, but these extend the analysis time.

Figure 2. Anton Paar SVM3000 viscometer is shown with Xsample 460 autosampler with the 48-vial carousel rack

Anton Paar sells two models of autosampler. The Xsample360 is better described as an autoinjector since it holds a single vial that must be placed by the operator. The Xsample 460 comes standard with a 48-position carousel rack (part no. 13559) that holds 12-mL vials. It is also available with a 24-position rack (part 13558) that holds 50-mL vials. It incorporates both automated injection and automated sample-changing. From a cleaning standpoint, the two units are functionally identical; the racks are irrelevant. They both work by a simultaneous application of pressure (via two diaphragm pumps) and suction (via piston pump) to the sample. A concentric needle-in-needle punctures the vial cap. At the same time that air is forced into the headspace from an outlet in the outer needle, suction is applied to the inner needle, drawing the fluid into the tubing, which has a volume of about 1 mL.

Obviously, flush volume is of greatest importance when the two fluids are dissimilar. Dissimilarity refers not only to the viscoelastic properties of the fluids, but also to their miscibilities and rate of mixing. The more unlike the two fluids are, the longer it takes for the residuum to become fully mixed with the second fluid and thus, the longer

it takes to fully wash out the first fluid. Moreover, there is always some residuum. The real issue is whether that residuum is of sufficient quantity that it contributes to the measurement process in a detectable manner and how to reduce the quantity so that the influence of the residuum becomes undetectable.

Table 4. Relative difference[a] in measured properties[b] from injection to injection, showing the effect of dilution of the residuum from the previous injection

Oil[c]	Set	Trial	η_∞ mPa s	μ_∞ mm^2 s^{-1}	ρ_∞ g cm^{-3}	T_∞ °C	$10^6 \times (P_n - P_{n-1})/P_{n-1}$ for η_∞	for μ_∞	for ρ_∞	for T_∞
RTO	a	1	27.439	28.316	0.9690	40.011				
RTO	a	2	26.374	27.035	0.9755	40.009	–38813	–45239	6708	–50
RTO	a	3	26.328	26.973	0.9761	40.011	–1744	–2293	615	50
PP30	a	1	81.312	93.840	0.8665	40.001				
PP30	a	2	83.299	96.187	0.8660	39.999	24437	25011	–577	–50
PP30	a	3	83.614	96.556	0.8660	39.998	3782	3836	0	–25
CGO	a	1	102.46	117.36	0.8730	39.998				
CGO	a	2	102.96	117.91	0.8732	39.998	4880	4686	229	0
CGO	a	3	103.32	118.33	0.8732	39.998	3497	3562	0	0
RTO	b	1	27.903	28.784	0.9694	40.008				
RTO	b	2	26.534	27.204	0.9754	40.008	–49063	–54881	6189	0
RTO	b	3	26.365	27.012	0.9760	40.010	–6369	–7069	615	50
CGO	b	1	100.30	114.79	0.8738	39.996				
CGO	b	2	103.08	118.33	0.8712	39.998	27717	30839	–2976	50
CGO	b	3	103.39	118.40	0.8732	39.998	3007	592	2296	0
PP30	b	1	84.053	97.008	0.8665	40.010				
PP30	b	2	84.267	114.39[d]	0.7369[d]	39.998	2546	NC[e]	NC[e]	–300
PP30	b	3	83.952	96.930	0.8661	40.007	–3738	–804[f]	–462[f]	–75[f]
PP30	b	4	83.803	96.779	0.8659	39.998	–1775	–1558	–231	–225
RTO	c	1	27.931	28.786	0.9703	40.008				
RTO	c	2	26.501	27.159	0.9758	40.008	–51198	–56521	5668	0
RTO	c	3	26.394	27.038	0.9762	40.008	–4038	–4455	410	0

Notes: (a) Relative difference = $(P_n - P_{n-1})/P_{n-1}$; n represents a trial with $(n - 1)$ representing the previous trial; values in the table are relative differences multiplied by 1 million for ease in reporting; value of 10000 = 1% relative difference. (b) Properties: η (dynamic viscosity in mPa s), μ (kinematic viscosity in mm^2 s^{-1}), ρ (density in g cm^{-3}), and T (temperature in °C); values listed are final values (i.e., time = ∞) as determined by the firmware's criteria for stability. (c) RTO = Royco Turbine Oil 555 (DOD-L-85734), PP30 = Pitt Penn 30 (MIL-PRF-2104G), CGO = CSD gear oil 75 (MIL-PRF-2105E). (d) Density value is suspect; therefore, kinematic viscosity value is also suspect; see text for details. (e) NC = not calculated due to outlier; see note d. (f) Relative differences were calculated between trials 1 and 3 since trial 2 was rejected as an outlier.

As Table 4 shows, the measured properties get closer with each subsequent measurement. For dynamic viscosity, the change is almost always the same direction, but smaller with each injection, suggesting that the difference is not due to indeterminate error, but rather due to bias being eliminated as residue from the first sample is eliminated. For density, this same trend is observed for all cases except one. The trend in kinematic viscosity tends to follow that of dynamic viscosity because the dynamic viscosity differences from trial to trial are so usually much greater than the density differences. In all cases, the relative differences between trials 2 and 3 are less than 1%, suggesting that an 18 mL wash (10 mL syringe filled twice) displaces enough of the previous sample to make the impact of the residuum undetectable. The largest difference (5.6% in μ) between injections 1 and 2 (8 and 18 mL wash, respectively) was observed after switching from 30-weight mineral oil (PP30/2104) to helicopter transmission fluid (RTO/85734). The second largest difference (5.5% in μ) between injections 1 and 2 was observed after switching from 75-weight gear oil

(CGO/2105) to RTO/85734. The third largest difference (–4.5% in μ) between injections 1 and 2 was observed after switching from RTO/85734 to PP30/2104. As expected, greater viscosity differences between samples means more flushing is needed to prevent carryover.

Of course, another way to look at the data involves comparing the average values after 2 injections (18 mL wash) with the average values after 3 injections (28 mL wash). As the residuum of the previous sample is further diluted, we expect the values to converge within the limit of the indeterminate error of the technique. The dynamic and kinematic viscosity results in Table 5 differ by no more than 0.6%; therefore, we conclude that carryover has been made undetectable (i.e., the residuum of previous sample has been reduced to the level where it does not affect the measurement in an observable manner). Table 5 supports this conclusion for both the results at the recommended truncated measurement (i.e., 60 s) and the final measurement based on the firmware's stability criteria.

Table 5. Agreement[a] of average[b] values of selected properties[c] measured on the SVM3000 between trials (injections) 2 and 3, demonstrating the efficacy of an 18 mL wash

Oil[d]	Time	Trial	$\bar{\eta}$	$(\bar{\eta}_3 - \bar{\eta}_2)/\bar{\eta}_2$	$\bar{\mu}$	$(\bar{\mu}_3 - \bar{\mu}_2)/\bar{\mu}_2$	$\bar{\rho}$
	s		mPa s	%	$mm^2\ s^{-1}$	%	$g\ cm^{-3}$
RTO	∞	2	26.47	–0.41	27.13	–0.46	0.9756
	∞	3	26.36		27.01		0.9761
PP30	∞	2	83.78	0	96.19[e]	0.58[e]	0.8660[e]
	∞	3	83.78		96.74		0.8661
CGO	∞	2	103.02	0.33	118.12	0.21	0.8722
	∞	3	103.36		118.37		0.8732
RTO	60	2	26.66	–0.39	27.21	–0.45	0.9782
	60	3	26.56		27.09		0.9787
PP30	60	2	84.42	0	96.68[e]	0.55[e]	0.8681[e]
	60	3	84.51		97.21		0.8680
CGO	60	2	103.82	0.31	118.59	0.21	0.8744
	60	3	104.14		118.84		0.8753

Notes: (a) Agreement is assessed by the relative difference. (b) Average is the arithmetic mean of 6 measurements (PP30 and CGO) or 9 measurements (RTO). (c) Properties: η (dynamic viscosity in mPa s), μ (kinematic viscosity in $mm^2\ s^{-1}$), and ρ (density in $g\ cm^{-3}$); values listed are either final values (time = ∞) as determined by the firmware's stability criteria or at 60 s. (d) RTO = Royco Turbine Oil 555 (DOD-L-85734), PP30 = Pitt Penn 30 (MIL-PRF-2104G), CGO = CSD gear oil 75 (MIL-PRF-2105E). (e) Calculations are based only on values from set a, trial 2, due to discordant density data in set b, trial 2.

The overall behavior is readily illustrated graphically. Figures 3 and 4 show the dynamic and kinematic viscosities, respectively, as a function of injection number for the synthetic helicopter transmission fluid (RTO). Note that the viscosity starts high with each set of injections. In both

cases, an oil with higher viscosity was the previous sample; consequently, each subsequent injection washes out more of the residuum, yielding lower and lower values. At 30 s, the rotor motion has not yet stabilized sufficiently to ensure precise dynamic viscosity determination as sets b and c show in Figure 3. In addition, there is a large positive bias because the rotor has not yet reached its maximum rotational velocity. This determinate error is nearly gone by 60 s. Figures 3 and 4 clearly show the asymptotic progression towards the "true" value as the residuum of the previous sample is diluted further by consecutive injections. By the third injection of the set, the difference between the two data is nearly indistinguishable from the random error of the measurement process.

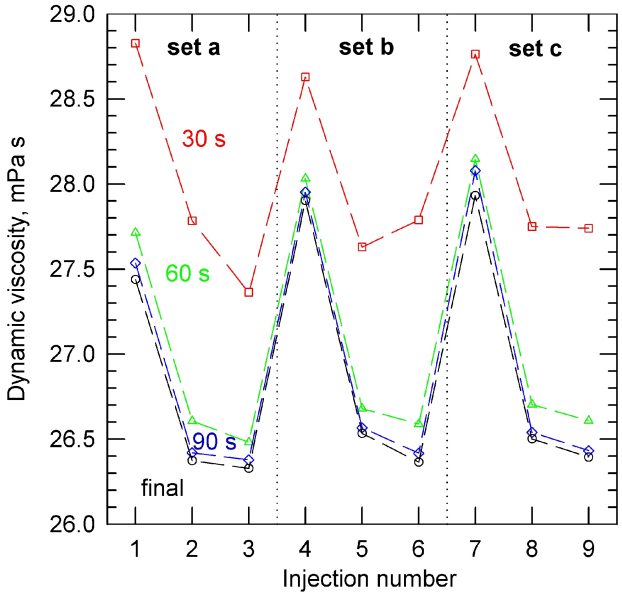

Figure 3. Dynamic viscosity varies with injection number for Royco Turbine Oil 555 (DOD-L-85734) on the SVM3000 at 40 °C. Immediately prior to set b, CSD Gear Oil 75 (MIL-PRF-2105E) was run. Immediately prior to set c, Pitt Penn 30 (MIL-PRF-2104G) was run. Each trace represents a measurement time: 30 s (red squares); 60 s (green triangles); 90 s (blue diamonds); final (black circles).

Alternatively, the same data may be presented as a function of stabilization time as has been done in Figures 5 and 6. Diminishing returns are realized because it takes increasingly longer for the viscosity to change as much. In other words, the improvement realized in going from 30 to 60 s is greater than that realized in going from 60 s to 90 s. In moving from 90 s to completion (final values were arbitrarily plotted at 2 minutes for convenience), the improvement may not eve be distinguishable from the indeterminate error associated with the technique. From the plots, it is clear that the difference in meaured viscosity that results from truncating at 60 s and allowing the full stabilization time is too small to be meaningful for the Army OAP needs. It is easier to see how the carryover from the previous sample influences the viscosities without regard to measurement duration in these two figures. Note that all the injection 1 plots are considerably higher than the injection 2 and 3 plots. Figures 5 and 6 also illustrate a statistically real difference between injections 2 and 3. However, the crux of the matter here is whether than difference matters for the decision-making progress. The red dotted line represents the maximum value obtained for any injection 2 at 60 s, and the blue dotted line represents the minimum value obtained for any injection 3 at completion.The difference between these two lines is the maximum error that would result from both taking the measurement at 60 s and using only two injections (18 mL flush volume) and it is about 0.28 mPa s for the dynamic viscosity, which is about 1% error, certainly acceptable for the programmatic needs of deciding whether to change the oil.

Similar results are seen if we plot the same kinds of graphs for one of the other oils. Figures 7-10 are the same types of presentations of the data for CSD Gear Oil 75 (MIL-PRF-2105E) as Figures 3-6 were for Royco Turbine Oil 555 (DOD-L-85734). The major difference we observe is that the first injection has a viscosity much lower than subsequent injections, which makes sense because gear

oil is much more viscous than transmission fluid or car engine oil. Moreover, when the transmission fluid preceded the gear oil, the first injection of gear oil gave a lower viscosity than when the 30-weight car engine oil preceded the gear oil. We also observe that the difference is sufficiently small between the viscosities found by the restricted procedure (60 s stabilization and 18 mL wash) and the "ideal" procedure (firmware stability criteria and 28 mL wash).

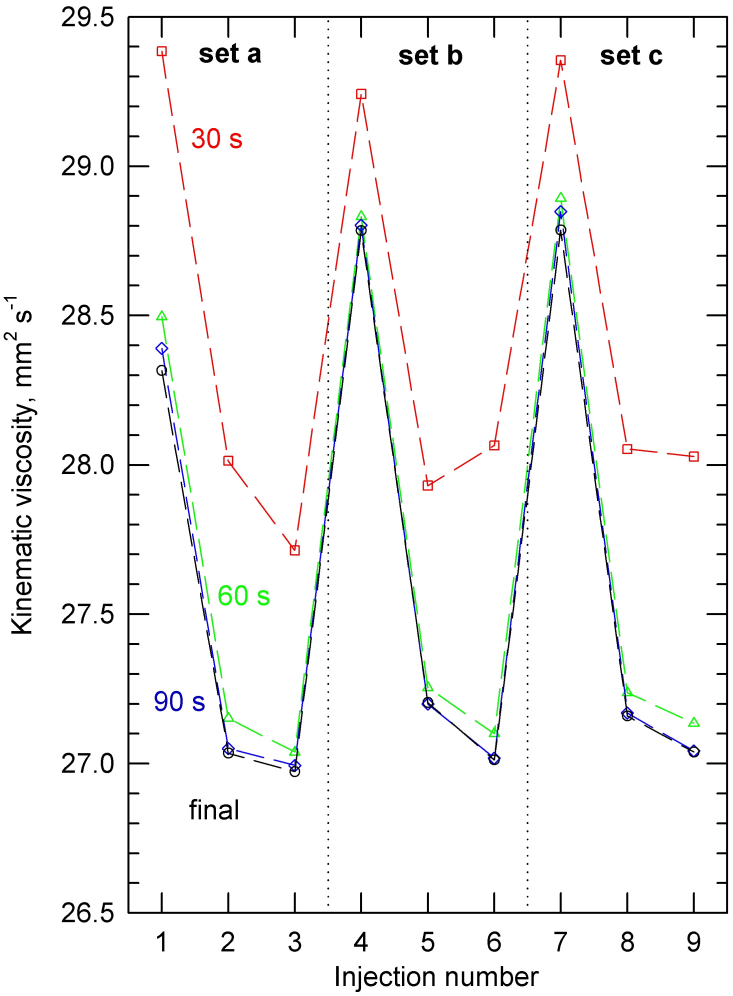

Figure 4. Kinematic viscosity varies with injection number for Royco Turbine Oil 555; see Figure 3 caption for more details.

In addition, to these experimental measures, we can *a priori* predict the maximal effect of each subsequent injection in diluting the previous sample's residuum by realizing that the worst case would be the complete mixing of the previous sample P and the newly injected sample N. If we assume volume is conserved, each time the same dilution factor is applied with 2 mL from the previous injection (the combined volume of the densimeter and rotor chamber) and 10 mL of newly injected sample. The fraction of P is then 2/12 (16.7% v/v) on the first injection. On the second injection, this is further diluted by the same dilution factor so that the fraction of P is substantially reduced: $(2/12)^2$ = 2.8% v/v. On the third injection, the dilution factor is again applied, so that the fraction of P is $(2/12)^3$ = 0.46% v/v. Certainly, there is no opportunity for complete mixing. Given the worst case: N_1 = 83.3%, N_2 = 97.2%, and N_3 = 99.5%, it is clear that two washes should be plenty.

If we consider the use of an autosampler, we must allow 1 mL for dead volume in the tubing. Even if the dilution factor now becomes 3 mL to 13 mL, the net impact remains similar; now $N_1 = 1 - (3/13) = 76.9\%$ v/v; $N_2 = 1 - (3/13)^2 = 94.7\%$ v/v; and $N_3 = 1 - (3/13)^3 = 98.8\%$ v/v. In other words, the worst case situation would have a sample that is 94.7% pure on the second injection. It is important to keep in mind that the difference in viscosity between any two samples is not that large relative to the viscosity itself, maybe ±15% at worst because the viscosity specifications for lubricating oils are so tight. Consequently, there will be a 5% contribution by something that varies potentially by 30% (+15% on one and –15% on the other), which is 1.5% (if we assume linear effects in admixtures). Only two conditions make this untrue: if the adjacent samples have vastly different viscosities or if they are not miscible (as coolant and truck/car engine oil are immiscible). Obviously, we would want to group oil samples by oil type and grade or weight to ensure the effect of residual sample is minimized.

3.4. Dealing with Outliers

A sample whose first test result shows it to be out of specification can be repeated to eliminate outliers. In fact, the outlier we observed can be used to illustrate this. Consider now the lone outlier of PP30 (set b, trial 2), which would have been treated as valid data in a single-run testing protocol. It represents a single occurrence where the density of the sample is believed to be discordant. Presumably, an air bubble in the U-tube densimeter led to a falsely low density determination. This low density, in turn, led to the calculation of an aberrantly high kinematic viscosity. The suspect value for μ was 114.3 $mm^2\ s^{-1}$, while $\bar{\mu}$ for the other three runs was 96.9 $mm^2\ s^{-1}$, a relative difference of 18%. A clear way to work around such outliers is to automatically reanalyze the sample whose first result suggests that it is out of specification. Since we witnessed only one such errant analysis for all of our runs, we estimate the rate of mechanical errors as 1 out of 21. Accordingly, the probability of two erroneous results for the same sample would be about 2.3%. On the other hand, anecdotal evidence suggests that errant injections are even rarer than we observed, so that the probability of two false readings is expected to be even lower.

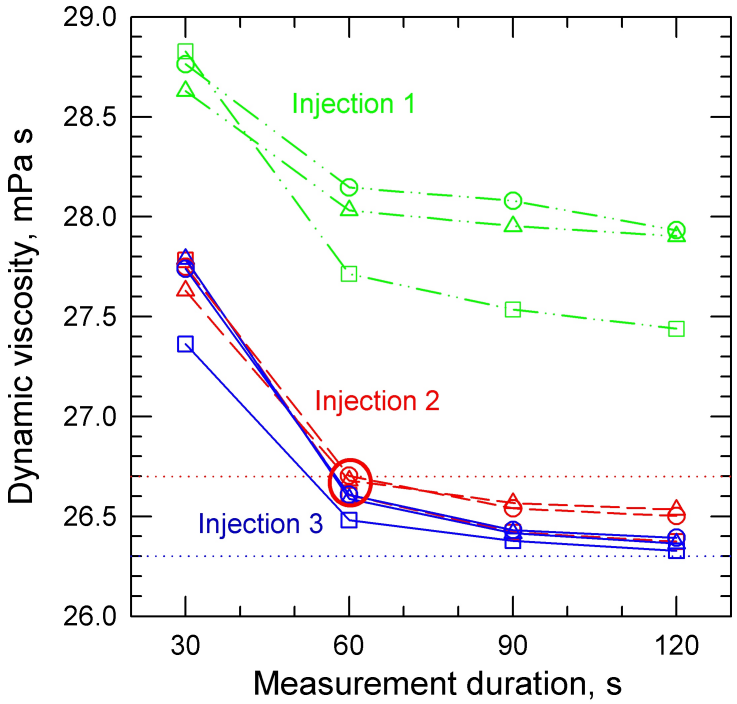

Figure 5. Dynamic viscosity varies with the time elapsed before taking the reading (stabilization time). Set is designated by shape: (a) squares, (b) triangles, (c) circles. Injection is shown by color: (1) green, (2) red, (3) blue. Final time was arbitrarily plotted at 120 s for convenience. Note that the first injection of any set always has error caused by carryover from the previous sample, but that this effect virtually disappears by the second injection. The red oval shows the values resulting from the operating parameters recommended in the Conclusions. The difference between the red and blue dotted lines shows the worst case error for any one value due to restricting both flush volume and stabilization time as recommended. Note the area where overlap occurs among the experimental viscosities, indicating that random error is now contributing significantly to the overall error. See Figure 3 caption for operating and sample details; this Figure is an alternate presentation of the same data.

On the other hand, it appears that by employing the dynamic viscosity as the standard of measurement, the rate of mechanical errors might be reduced.The dynamic viscosity of the injection (no. 2) with the discordant density was 40.2 mPa s as was the arithmetic mean of the other three replicate injections (nos. 1, 3, and 4). In other words, there was no difference; the dynamic viscosity found for this injection was correct. During this investigation, one less sample (saved time) would have been required. If this had been a real life oil sample and it had not been rerun, there could have been an unnecessary oil change (wasting material and labor and incurring disposal cost). Therefore, it is again recommended that the dynamic viscosity be adopted in favor of the kinematic viscosity as a measure of oil quality. If a separate specification on the density were to be put in place, there would perhaps be some potential value to having any two quantities that incorporate both the dynamic viscosity and the density: (η, ρ), (η, μ), or (μ, ρ). Right now, though, no explicit or reliable implicit density specification exists that mandates an oil change. This is an important problem

because water contamination increases both η and ρ, but fuel tends to decrease both η and ρ. Since μ = η /ρ, it is possible for the effects to cancel one another out so that μ remains relatively constant even though both η and ρ have changed.

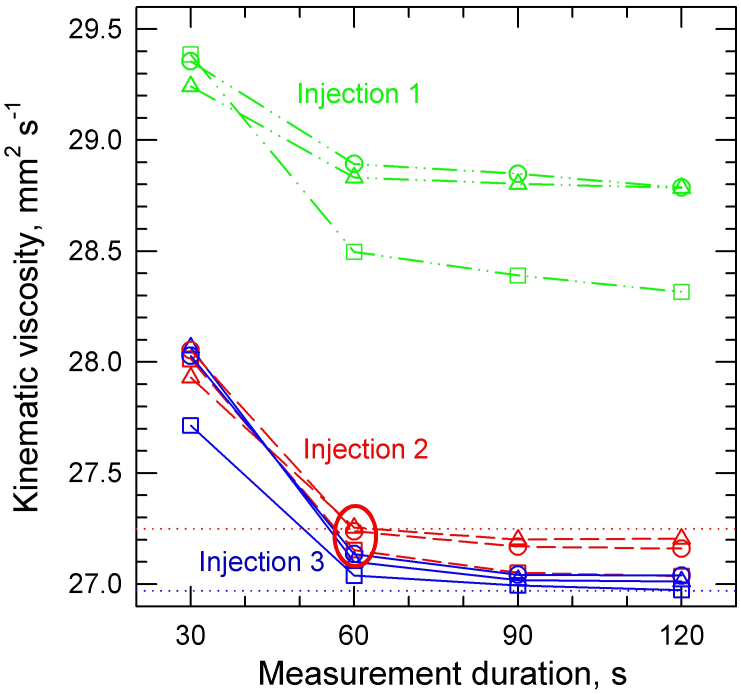

Figure 6. Kinematic viscosity varies with time elapsed prior to taking a reading (stabilization time). See Figure 5 caption for key to notation and operating conditions.

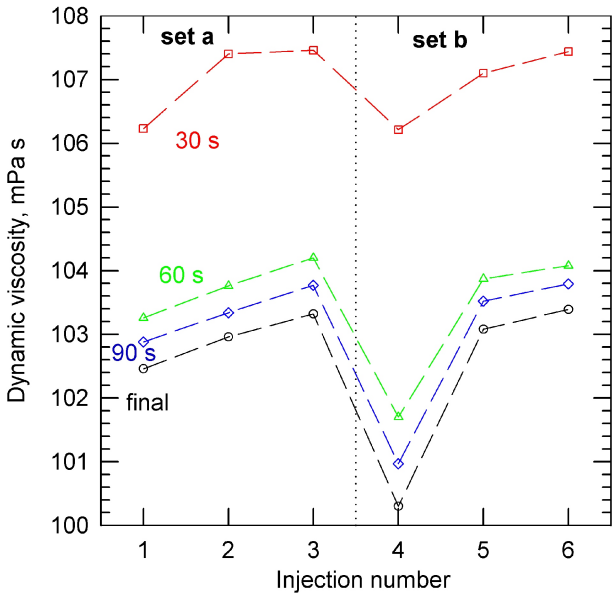

Figure 7. Dynamic viscosity of CSD Gear Oil 75 (MIL-PRF-2105E) varies with injection number (wash volume). Each trace represents a measurement duration (stabilization time): 30 s (red squares); 60 s (green triangles); 90 s (blue diamonds); final (black circles). Set a was immediately preceded by PP30, while set b was immediately preceded by RTO. As expected, the residuum from the previous sample causes the first injection to be low. The RTO, whose viscosity is the lowest of all three types, induces a lower first-injection viscosity. Also note that 30 s is not enough time to see this effect. This may be due to a combination of incomplete mixing and incomplete quasi-equilibration of the viscous and drag forces.

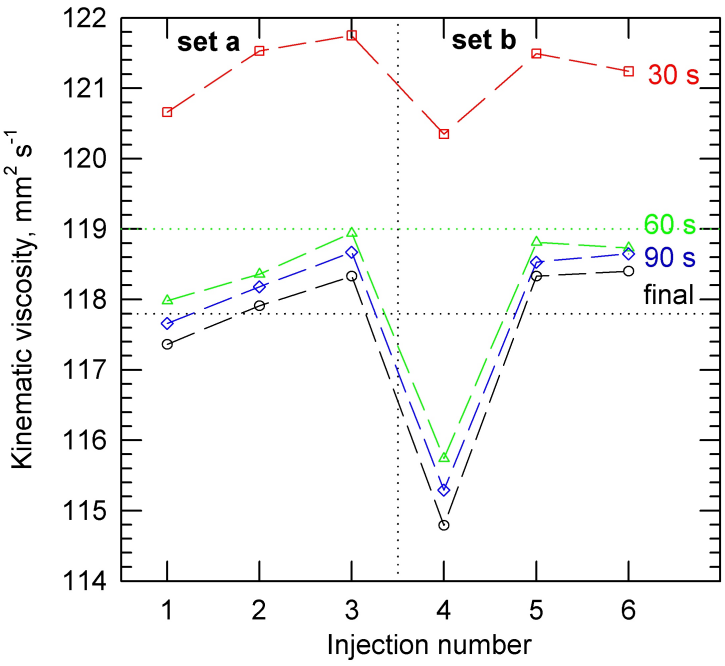

Figure 8. Kinematic viscosity varies with injection number (flush volume) for CSD Gear Oil 75. See Figure 7 for more details. The space between the horizontal lines represents the difference between the "ideal" (i.e., third injection and final value) and restricted (i.e., second injection and 60 s). Although the space between the two lines appears large, it translates to 1% error in the kinematic viscosity, which exceeds the requirements for Army OAP decision-making.

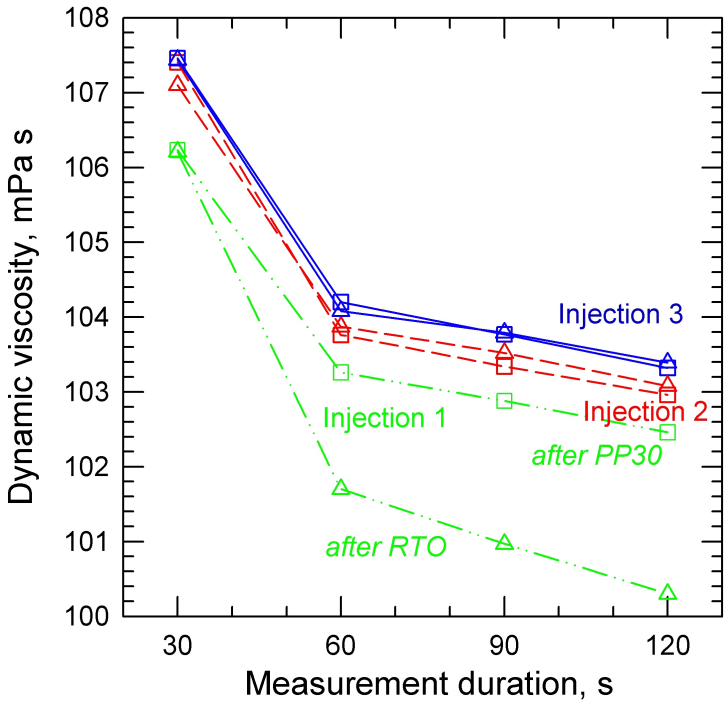

Figure 9. Dynamic viscosity of CSD Gear Oil 75 varies with time elapsed prior to taking the reading (stabilization time). This is an alternative presentation of the data in Figure 7. Set is designated by shape: (a) squares, (b) triangles. Injection is designated by color: (1) green, (2) red, (3) blue. Final time was arbitrarily plotted at 120 s for convenience. Note that the first injection is always biased low due to carryover of either RTO (lower) or PP30. Also note the closeness of viscosities for injection 2 at 60 s and those for injection 3 at final stabilization. The error caused by the restricted procedure is less than 1%. Compare and contrast with Figures 5 and 7.

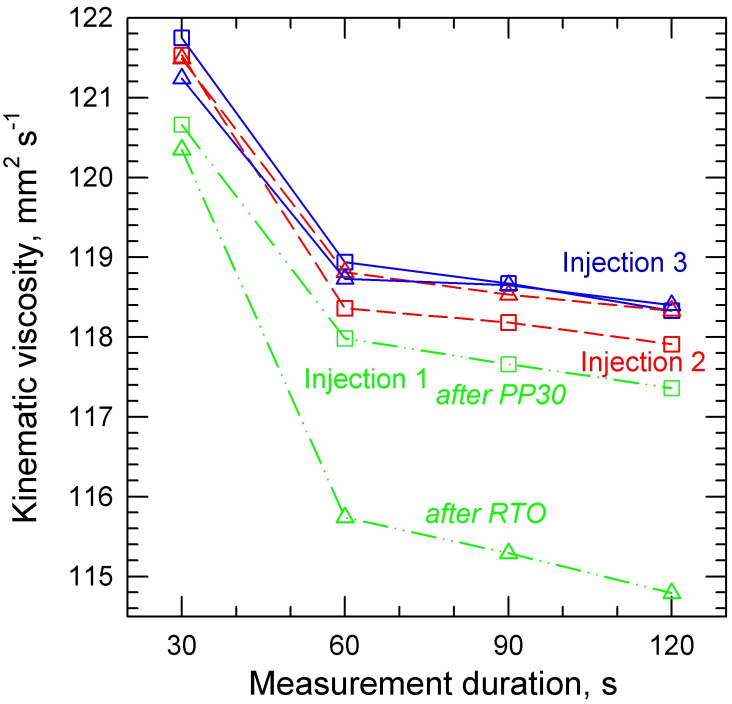

Figure 10. Kinematic viscosity varies with time elapsed prior to taking a reading (stabilization time). See Figure 7 for details. This is an alternative presentation of the data in Figure 8. Kinematic viscosity behavior closely follows dynamic viscosity behavior of Figure 9 due to tightness of density measurements. See Figure 9 caption for key to notation and operating conditions as well as additional explanation. Compare and contrast with Figures 6 and 8.

4. Conclusions

4.1. ASTM D 7042-04 Suitability

The rigors of ASTM standard D 7042-04 exceed the programmatic requirements of the Army OAP. The standard biases the golden triangle (good, fast, cheap) in favor of higher data quality and sacrifices the competing interests of cost and speed. Although this ASTM standard is applicable to the measurement, a less rigorous approach is certainly more cost-effective.

4.2. Dynamic Versus Kinematic Viscosity

Use of dynamic viscosity is preferred over kinematic viscosity in that it reduces opportunities for mechanical errors during injection and is less likely to be confounded by phenomena that affect both

dynamic viscosity and density similarly (e.g., fuel and water). Unless an independent explicit or implicit density criterion is instituted for oil quality, information is lost by reliance on the kinematic viscosity, which convolves dynamic viscosity and density.

4.3. Measurement Duration (Stabilization Time)

Measurement duration (stabilization time) can be set to 60 seconds while producing data of quality sufficient to meet Army OAP programmatic needs. The extant firmware does not permit this.

4.4. Flush Volume

Flush volumes of 20.0 mL will satisfactorily purge and recondition the densimeter and viscometer measuring chamber—without washes between the analyses. This practice yields data of sufficient quality for Army OAP decision-making. This requires the 24-position carousel (part no. 13558) for the Xsample 460, which uses a 50 mL sample vial.

4.5. SVM3000 Manual Operation

Manual operation of an SVM series Stabinger viscometer can achieve or exceed the required Army OAP throughput (40 samples per hour). This is done by drawing the sample into a 10 mL syringe and injecting it twice. The last portion of the second injection is tested.

4.6. SVM3000 Firmware Limitations and Revisions

An autosampler-equipped SVM series Stabinger viscometer is unable to achieve the required Army OAP throughput with the current firmware configuration. Nevertheless, a revised configuration (currently under development) is anticipated to solve the problem. Since the time that we began this study, Anton Paar has released a new version of the firmware that permits the user to set the inter-sample washes to zero. It has not yet created an option for fixed time measurement.

Anton Paar reports that it is expanding the options in its firmware to provide the flexibility for users who do not require the rigor of ASTM D7042-04. Furthermore, knowledge of the samples' miscibilities allows us to conclude that the wash and dry cycles are unrequired, whereas ASTM D 7042-04 was set up to be as rugged and robust as possible without regard to inter-sample miscibility. There is very little, if any, reason to permit more than 60 seconds to elapse for stabilization. To do so would not improve the results meaningfully and would preclude the possibility of attaining the desired throughput of 40 samples per hour. Nonetheless, the current configuration of the firmware proscribes this option if the autosampler is to be used. When the samples are injected manually and the measurement is taken manually, the firmware is not employed. In its present form, the Stabinger viscometer and its firmware cannot achieve the desired throughput, but this is an artefact of the configuration rather than a restriction of the science.

5. References

(1) Urbansky, E.T.; Poff, M.L.; Taylor, C.L. *Evaluation of three commercial viscometers*. JOAP TSC; Pensacola, FL; June 7, 2004; JOAP-TSC-TR-04-01.

(2) Novotny-Farkas, F.; Böhme, W.; Stabinger, H.; Belitsch, W. The Stabinger viscometer–a unique new instrument for oil service laboratories. World Tribology Congress II, 2001.

(3) ASTM D 7042-04. Standard Test Method for Dynamic Viscosity and Density of Liquids by Stabinger Viscometer (and the Calculation of Kinematic Viscosity). American Society for Testing and Materials; West Conshohocken, PA.

(4) Personal communication. Daniel McElroy, U.S. Army Oil Analyis Program, Logistics Support Agency, Army Materiel Command, Redstone Arsenal, AL, to one of the authors (ETU); June 2006.

Appendix 1

Raw data for physical properties[a] reported by the Anton Paar SVM3000 for individual injections recorded at various measurment times[b]

		30 s				60 s			
Oil[c]	Trial	η mPa s	μ $mm^2\ s^{-1}$	ρ $g\ cm^{-3}$	*T* °C	η mPa s	μ $mm^2\ s^{-1}$	ρ $g\ cm^{-3}$	*T* °C
RTO	1	28.827	29.385	0.9740	40.173	27.713	28.496	0.9713	40.011
	2	27.783	28.014	0.9813	40.170	26.606	27.152	0.9784	40.027
	3	27.363	27.714	0.9811	40.173	26.482	27.038	0.9784	39.996
PP30	1	86.731	98.627	0.8712	40.194	82.512	94.938	0.8688	39.994
	2	87.433	99.489	0.8702	40.175	84.026	96.678	0.8681	39.977
	3	88.456	100.460	0.8705	40.187	84.428	97.179	0.8682	39.987
CGO	1	106.23	120.66	0.8775	40.161	103.26	117.98	0.8753	39.972
	2	107.40	121.53	0.8775	40.163	103.76	118.36	0.8754	39.972
	3	107.46	121.75	0.8774	40.170	104.20	118.94	0.8754	39.991
RTO	1	28.630	29.242	0.9742	40.150	28.032	28.831	0.9716	40.010
	2	27.629	27.931	0.9806	40.167	26.680	27.254	0.9778	40.003
	3	27.788	28.065	0.9817	40.163	26.588	27.100	0.9787	40.029
CGO	1	106.21	120.35	0.8786	40.198	101.70	115.74	0.8763	39.996
	2	107.10	121.49	0.8753	40.168	103.87	118.81	0.8733	39.969
	3	107.44	121.24	0.8772	40.139	104.08	118.73	0.8752	39.970
PP30	1	89.211	101.39	0.8710	40.184	84.792	97.559	0.8685	40.003
	2	88.415	117.52[d]	0.7442[d]	40.155	84.822	114.37[d]	0.7404[d]	39.983
	3	87.746	100.85	0.8702	40.162	84.601	97.247	0.8678	39.983
	4	86.751	98.788	0.8693	40.113	84.232	97.056	0.8677	39.970
RTO	1	28.763	29.355	0.9756	40.158	28.146	28.892	0.9725	40.010
	2	27.750	28.053	0.9810	40.155	26.702	27.238	0.9783	40.027
	3	27.740	28.028	0.9819	40.143	26.608	27.135	0.9790	40.041

continued on next page

Appendix 1 continued

		30 s				60 s			
Oil	Trial	η mPa s	μ $mm^2\ s^{-1}$	ρ $g\ cm^{-3}$	*T* °C	η mPa s	μ $mm^2\ s^{-1}$	ρ $g\ cm^{-3}$	*T* °C
RTO	1	27.535	28.390	0.9699	39.965	27.439	28.316	0.9690	40.011
	2	26.420	27.051	0.9766	39.969	26.374	27.035	0.9755	40.009
	3	26.378	26.994	0.9770	39.972	26.328	26.973	0.9761	40.011
PP30	1	81.834	94.346	0.8676	39.970	81.312	93.840	0.8665	40.001
	2	83.665	96.438	0.8670	39.972	83.299	96.187	0.8660	39.999
	3	83.936	96.764	0.8670	39.964	83.614	96.556	0.8660	39.998
CGO	1	102.88	117.66	0.8741	39.972	102.46	117.36	0.8730	39.998
	2	103.34	118.18	0.8743	39.974	102.96	114.91	0.8732	39.998
	3	103.77	118.67	0.8743	39.976	103.32	118.33	0.8732	39.998
RTO	1	27.953	28.803	0.9702	39.972	27.903	28.784	0.9694	40.008
	2	26.567	27.200	0.9764	39.969	26.534	27.204	0.9754	40.008
	3	26.416	27.018	0.9772	39.965	26.365	27.012	0.9760	40.010
CGO	1	100.97	115.29	0.8750	39.962	100.30	114.79	0.8738	39.996
	2	103.52	118.53	0.8722	39.972	103.08	118.33	0.8712	39.998
	3	103.79	118.65	0.8742	39.977	103.39	118.40	0.8732	39.998
PP30	1	84.353	97.205	0.8673	39.967	84.053	97.008	0.8665	40.010
	2	84.400	114.36[d]	0.7387[d]	39.970	84.267	114.390[d]	0.7369[d]	39.998
	3	84.115	97.126	0.8669	39.970	83.952	96.930	0.8661	40.007
	4	84.091	96.976	0.8668	39.977	83.803	96.779	0.8659	39.998
RTO	1	28.079	28.848	0.9712	39.969	27.931	28.786	0.9703	40.008
	2	26.539	27.169	0.9769	39.969	26.501	27.159	0.9758	40.008
	3	26.431	27.042	0.9774	39.969	26.394	27.038	0.9762	40.008

Notes: (a) Properties: η (dynamic viscosity in mPa s), μ (kinematic viscosity in $mm^2\ s^{-1}$), and ρ (density in g cm^{-3}). (b) Values listed are either final values (time = ∞) as determined by the firmware's stability criteria or at 30, 60, or 90 s as indicated. (c) RTO = Royco Turbine Oil 555 (DOD-L-85734), PP30 = Pitt Penn 30 (MIL-PRF-2104G), CGO = CSD gear oil 75 (MIL-PRF-2105E). (d) Suspect density data attributed speculatively to air bubble in densimeter. Because kinematic viscosity is the ratio of dynamic viscosity to density, it is also suspect. PP30 trial 2 densities and kinematic viscosities for set b were all discarded as outliers.

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